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(54) SILICONE RUBBER COMPOSITION FOR WIPER BLADE**(57)Abstract:**

PROBLEM TO BE SOLVED: To obtain a silicone rubber composition for wiper blades, which yields windshield wiper blades in general, especially a silicone wiper blade which has a good sliding characteristic against any glass surface and a sufficient tear strength.

SOLUTION: This composition essentially comprises (A) 100 pts.wt. organopolysiloxane of the formula: $R_nSiO(4-n)/2$ (wherein each R is identical to or different from each other and is a non-substituted or substituted monovalent hydrocarbon group; and (n) is a positive number of from 1.98 to 2.0) having at least two alkenyl groups in one molecule, (B) from 10 to 100 pts.wt. reinforcing silica having a specific surface area of 50 m²/g or larger, (C) from 10 to 200 pts.wt. diatomite, (D) from 0.1 to 30 pts.wt. of an aromatic polyamide fiber and (E) a hardener at an amount effective for curing.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] Generally this invention relates to the silicone rubber constituent which gives a windshield wiper blade and the silicone wiper blade which has good slipping nature to any glass sides, and has sufficient tear strength especially.

[0002]

[Description of the Prior Art] Conventionally, as wye PABU rate material, casts which carried out chloridization of the front face of natural rubber or natural rubber, such as a thing and synthetic rubber, have been used. However, in such rubber molding, the agglutination phenomenon happened between the glass side and the wiper blade by use in the time of half-dryness, or a cold district, or rubber elasticity was lost for low temperature, the problem which does not function as a wiper arose and the so-called "chatter phenomenon" had occurred. Therefore, it excels in cold resistance, and the wiper-blade material using the silicone rubber which can be colored is proposed, for example, molybdenum disulfide is added to silicone rubber in JP, 57-2940, B. In the thing and JP, 5-117530, A which gave slipping nature, to silicone rubber, diatomaceous earth, Although what was made to contain parallel cleavage mineral powder and fluororesin powder, and improved the chatter phenomenon, the thing to which the nature bulking agent of a silica was added in JP, 4-154258, A, and coefficient of friction to a glass side was reduced are known By adding the bulking agent for all raising the slipping nature and wiping nature to a glass side, the tear strength as a wiper blade cannot fall and endurance sufficient in a mounting examination on a bad road cannot be discovered. Moreover, although the wiper-blade material which used the high modulus quantity tear rubber compound was illustrated in JP, 1-249858, A, since the slipping nature to a glass side was bad, it was what does not bear use.

[0003] this invention was made in order to improve the above-mentioned situation, and it aims at offering the silicone rubber constituent for wiper blades which has good slipping nature and gives the high wiper blade of a tear strength.

[0004]

[A The means for solving a technical problem and the form of implementation of invention] In order that this invention persons may attain the above-mentioned purpose. The organopolysiloxane which has at least two ARUKENIRU machines in the (A) molecule, and is expressed with the following average empirical formula (1) as a result of inquiring wholeheartedly, (B) by blending (C) diatomaceous earth and (D) aromatic-polyamide fiber with the silicone rubber constituent containing the reinforcement nature silica more than specific-surface-area of 50m²/g, and the (E) curing agent It finds out that the silicone rubber constituent which gives the silicone rubber which was excellent as wiper-blade material, and which wipes off and has a performance, abrasion resistance, endurance, and cold resistance is obtained, and came to make this invention.

[0005] Therefore, this invention has at least two ARUKENIRU machines in the (A) molecule, and is the following average empirical formula (1).

$R_nSiO(4-n)/2$ (1) (the monovalent hydrocarbon group of the same or of a different kind un-replacing or substitution is shown by the inside R of a formula, and n is the positive number of 1.98-2.0.)

The organopolysiloxane come out of and expressed The reinforcement nature silica more than 100 weight sections (B) specific-surface-area of 50m²/g 10 - 100 weight section (C) diatomaceous earth 10 - 200 weight (section D) aromatic-polyamide fiber 0.1 - 30 weight section (E) curing agent Hardening effective dose. The silicone rubber constituent for wiper blades characterized by considering as an indispensable component is offered.

[0006] Hereafter, this invention is explained in detail. (A) The organopolysiloxane of a component has at least two alkenyl machines in a molecule, and is the following average empirical formula (1).

$R_nSiO(4-n)/2$ (1) (the monovalent hydrocarbon group of the same or of a different kind un-replacing or substitution is shown by the inside R of a formula, and n is the positive number of 1.98-2.0.) It is shown.

[0007] In this case, it is R. Cycloalkyl machines, such as alkyl groups, such as **, a methyl group, an ethyl group, a propyl group, and a butyl, and a cyclohexyl machine, Alkenyl machines, such as a vinyl group, an allyl group, a butenyl group, and a hexenyl machine, Aralkyl machines, such as aryl groups, such as a phenyl group and a tolyl group, a benzyl, and beta-phenylpropyl machine, A part or all of a hydrogen atom that was combined with the carbon atom of these bases Or a halogen atom, it is chosen from the chloro methyl group replaced by the cyano group etc., a truffle RUORO propyl group, a cyano ethyl group, etc. -- the same or different species -- desirable -- carbon numbers 1-12 -- the monovalent hydrocarbon group of un-replacing [of carbon numbers 1-8] or substitution is mentioned more preferably Moreover, n should be the positive number of 1.98-2.02, and although the chain end should be blocked by a trimethylsilyl machine, the dimethyl vinyl

silyl machine, the dimethyl hydroxy silyl machine, the methyl divinyl silyl machine, the trivinylsilyl group, etc., set this organopolysiloxane to this invention. This organopolysiloxane needs to have at least two ARUKENIRU machines in a molecule, and it is [0.001-10 mol %, especially 0.01-5 mol% of organopolysiloxane] desirable among R an ARUKENIRU machine and that it is especially a vinyl group. Moreover, the viscosity of viscosity in 25 degrees C is [the thing of 100 or more cses] desirable. It is 100,000-10,000,000cs more preferably. As for especially polymerization degree, 3,000 or more are [100 or more] desirable, and an upper limit is 100,000 and is 10,000 more preferably.

[0008] Next, the reinforcement nature silica of the (B) component is used for the purpose which obtains the silicone rubber which was excellent in the mechanical strength. Although a dry type silica or a wet silica is sufficient as these silicas as long as specific surface area is the thing of 100-400m²/g preferably more than 50m²/g, in order to obtain intensity, a dry type silica (mist silica) is desirable. Moreover, you may use the silica by which surface treatment was carried out by organopolysiloxane, ORGANO polysilazane, the chlorosilicane, alkoxyasilane, etc. in the front face if needed. in addition -- since processability will become remarkably bad and the intensity of the obtained silicone rubber will also fall, if it is too few in under 10 weight sections, and the effect of sufficient reinforcement nature does not show up to the organopolysiloxane 100 weight section of the (A) component but the addition of this reinforcement nature silica exceeds the 100 weight sections -- the 10 - 100 weight section -- it is 30 - 80 weight section preferably

[0009] In this invention, in order to give the slipping nature to the glass front face of a windshield as a (C) component, diatomaceous earth is used. In this case, it is desirable to use together the diatomaceous earth with which at least two kinds of particle size differs. By using together two or more kinds from which a mean particle diameter differs, slipping nature improves remarkably. As for the mean particle diameter of the diatomaceous earth (I) which is especially one side, it is especially desirable that 100 micrometers or less of mean particle diameters of the diatomaceous earth (II) of 1-4 micrometers and another side are 50 micrometers or less especially exceeding 5 micrometers exceeding 5 micrometers 0.01-5 micrometers.

[0010] The loadings of the above-mentioned diatomaceous earth are the 10 - 200 weight section, especially the 20 - 100 weight section in total to the organopolysiloxane 100 weight section of the (A) component. When using together the diatomaceous earth with which at least two kinds of particle size differs, it is desirable as a weight ratio 90:10-10:90, and to set especially the rate of the above-mentioned diatomaceous earth (I) and diatomaceous earth (II) to 80:20-20:80, and, as for the addition of diatomaceous earth (I), it is especially desirable to the organopolysiloxane 100 weight section of the (A) component the 5 - 100 weight section and that it is 10 - 50 weight section. On the other hand, as for the addition of diatomaceous earth (II), it is especially desirable to the organopolysiloxane 100 weight section of the (A) component the 10 - 100 weight section and that it is 15 - 50 weight section.

[0011] In addition, each can carry out surface treatment of these diatomaceous earth if needed. As coupling agent, various chlorosilicanes, alkoxyasilane, a silazane, a carbon functional silane, a silicone oil, etc. are used.

[0012] Next, the aromatic-polyamide fiber of the (D) component is the component which raises the tear intensity of a silicone rubber hardened material, and is required to raise the endurance as a wiper blade, and abrasion resistance especially.

[0013] In this case, as aromatic-polyamide fiber, the aramid fiber which has the structure of the Para type, a meta-mold, and the Para-meta-compound die is suitable. For example, the Para type aramid (PORIPARA phenylene terephthalamide), a meta-mold aramid (polymeter phenylene iso phthalamide), and the Para-METAKO polyamide (a KOPORI phenylene, 3, 4-oxydiphenylene terephthalamide) are mentioned, and the Kevlar by the Du Pont make and Toray Industries, Inc., Conex by Teijin, Ltd., TEKUNORA, etc. are specifically used suitably. Although especially the fiber configuration of the above-mentioned aromatic-polyamide fiber is not restricted, for example, things, such as filament yarn, a chopped fiber, a stay bull fiber, pulp, and spanner IZUTO yarn, can be used, a chopped fiber is used suitably especially.

[0014] Moreover, if it may be unable to mix enough if fiber length is too long when especially especially 1-3 deniers and fiber length have 1-6mm desirable fiber 0.1-10mm and carry out addition mixture at a silicone rubber compound, and the diameter of fiber of aromatic-polyamide fiber is too short 0.05-10 deniers, sufficient tear intensity may not come out of it.

[0015] Each can carry out surface treatment of these aromatic-polyamide fiber if needed. As coupling agent, various chlorosilicanes, alkoxyasilane, a silazane, a carbon functional silane, a silicone oil, etc. are used.

[0016] the addition of aromatic-polyamide fiber -- the organopolysiloxane 100 weight section of the (A) component -- receiving -- 0.1 - 30 weight section -- it is 1 - 10 weight section preferably If there are too few additions, sufficient tear intensity will not be discovered, but if many [too], it is unmixable with silicone rubber to homogeneity.

[0017] (F) In case the hydrophilic grant agent of a component shifts to a dry shell semi sentiment and a wet state, it is desirable to add in order to assist the slipping nature of a wiper blade and a windshield. The silicone oil to which conversion of the hydrophilic properties, such as polyether conversion, sulfonyl conversion, carboxylic-acid conversion, and amine conversion, was carried out by the functional group to which it is made to give is suitably used in a part of polyether, such as polyethylene glycol, and silicone oil the making a hydrophilic property give purpose. The silicone oil which carried out polyether conversion preferably especially is used suitably. To the organopolysiloxane 100 weight section of the (A) component, preferably, it is 1 - 10 weight section more preferably, if fewer than the 0.01 weight sections, sufficient hydrophilic property will not be assisted, but an addition has 0.01 - 50 weight section and a possibility that the mechanical strength of silicone rubber may fall remarkably and weatherability may also fall if 50 weight sections are exceeded.

[0018] (E) As a curing agent of a component, organic peroxide or the curing agent by addition bridge formation is used. As organic peroxide. **, benzoyl peroxide, 2, 4-dichlorobenzoyl peroxide, p-methyl benzoyl peroxide, o-methyl benzoyl peroxide, 2, 4-dicumyl peroxide, 2, a 5-dimethyl-screw (2, 5-tert-butyl peroxide) hexane, G t-butyl peroxide, t-butyl par

benzoate, 1, and 1-screw (tert-butyl peroxide) - 3, 3, a 5-trimethyl cyclohexane, Organic peroxide, such as 1, 6-screw (tert-butyl peroxide carboxy) hexane, dicumyl peroxide, and cumyl-t-butyl peroxide, is used. These organic peroxide may be used independently and may use two or more sorts together. the addition of these organic peroxide -- the organopolysiloxane 100 weight section of the (A) component -- receiving -- 0.1 - 10 weight section -- it is 0.3 - 5 weight section preferably Even if bridge formation of under the 0.1 weight section is inadequate and it exceeds 10 weight sections, improvement in a cure rate cannot be desired.

[0019] As a curing agent by addition bridge formation, the ORGANO hydrogen polysiloxane and an addition-reaction catalyst are used. As the ORGANO hydrogen polysiloxane (although Rin formula' can show the monovalent hydrocarbon group of the same or of a different kind un-replacing or substitution and can mention the specifically same monovalent hydrocarbon group as what was illustrated by Above R, it does not include an aliphatic unsaturated bond preferably.) $R^aHbSiO(4-a-b)/2$ (2) Moreover, a and b are numbers with which are satisfied of $0 \leq a < 4$, $0 < b < 4$, and $0 < a+b < 4$. What is expressed can be used.

[0020] Specifically, it is as an ORGANO hydrogen polysiloxane. Both-ends trimethylsiloxy machine blockade methyl hydrogen polysiloxane, A both-ends trimethylsiloxy machine blockade dimethylsiloxane methyl hydrogen siloxane copolymer, Both-ends dimethyl hydrogen siloxy machine blockade dimethylpolysiloxane, A both-ends dimethyl hydrogen siloxy machine blockade dimethylsiloxane methyl hydrogen siloxane copolymer, A both-ends trimethylsiloxy machine blockade methyl hydrogen siloxane diphenyl siloxane copolymer, A both-ends trimethylsiloxy basis blockade methyl hydrogen siloxane diphenyl siloxane dimethylsiloxane copolymer, (CH₃) The copolymer which consists of the copolymer and 2(CH₃)HSiO_{1/2} unit which consist of 2HSiO_{1/2} unit and SiO_{4/2} unit, SiO_{4/2} unit, and 3(C₆H₅) SiO_{3/2} unit is mentioned.

[0021] It is desirable to the organopolysiloxane 100 weight section of the (A) component 0.1 - 30 weight section and to make especially the loadings of this ORGANO hydrogen polysiloxane into 0.3 - 10 weight section.

[0022] As an addition-reaction catalyst, platinum system catalysts, such as platinum system catalysts, such as a reactant of platinum black, the 2nd platinum of chlorination, a chloroplatinic acid, a chloroplatinic acid, and monohydric alcohol, a complex of a chloroplatinic acid and olefins, and a platinum screw acetoacetate, a palladium system catalyst, and a rhodium system catalyst, are mentioned. In addition, although the loadings of this addition-reaction catalyst can be made into the amount of catalysts, it is usually 10-5,000 ppm to the organopolysiloxane of the (A) component as a platinum metal.

[0023] In addition to the above-mentioned component, it responds to the constituent of this invention at the need, and is as various additives, such as bulking agents, such as carbon black, such as un-reinforcing nature silicas, such as a hydroxy-group content ORGANO siloxane, a trituration quartz, and a crystalline silica, acetylene black, furnace black, and channel black, and a calcium carbonate, a coloring agent, a tear on-the-strength improver, a heat-resistant improver, a fire-resistant disposition top agent, carrier acid, and a thermal conductivity improver, and a release agent or the dispersant for bulking agents. It is arbitrary to add various alkoxysilane especially phenyl group content alkoxysilane and its hydrolyzate, a diphenyl silane diol, a carbon functional silane, a silanol-group content low-molecular siloxane, etc.

[0024] The silicone rubber constituent of this invention can mix the above-mentioned component uniformly using rubber kneading machines, such as 2 rolls, a Banbury mixer, and a dough mixer (kneader), and can obtain it by heat-treating if needed.

[0025] Thus, the obtained silicone rubber constituent can obtain a silicone rubber wiper easily by carrying out heat hardening. That what is necessary is just the method to which sufficient heat for disassembly of a curing agent and vulcanization of silicone rubber is applied, the hardening method also extrudes the molding method, and is not restricted [foaming / mold / by injection / the continuous vulcanization by molding, a press,] especially. Moreover, you may vulcanize secondarily at 150-250 degrees C for about 1 to 10 hours if needed.

[0026] In addition, as for the silicone rubber constituent of this invention, it is desirable that the tear strengths of the silicone rubber which hardens this and is obtained are two or more 30 kgf/cm in the measurement based on A type (JIS 6301).

[0027]

[Example] Although an example and the example of comparison are shown and this invention is explained concretely hereafter, this invention is not restricted to the following examples. The performance as a wiper blade is shown in Table 1.

[0028] As the fumed silica (product made from Japanese Aerosil) 30 weight section the organopolysiloxane 100 weight section whose average degree of polymerization it consists of 0.025 mol % of 99.825 mol siloxane [0.15 mol % / of % and methyl vinyl siloxane units / and dimethyl vinyl] units of [examples 1-3] dimethylsiloxane units, and is about 8,000, and whose specific surface area are 300m²/g, and a dispersant. It has a both-ends silanol group, the dimethylpolysiloxane 10 weight section whose viscosity in the average degree of polymerization of 13 or 25 degrees C is 15cs(es) was kneaded by the kneader, it heat-treated at 180 degrees C for 3 hours, and the base compound 1 was prepared.

[0029] To the above-mentioned base compound 1, the diatomaceous earth 15 weight section whose mean particle diameter is 3 micrometers, and the diatomaceous earth 20 weight section whose mean particle diameter is 6 micrometers are added with 2 rolls, and it is a hydrophilic grant agent further. 1.5 weight section addition of the polyether conversion dimethylsiloxane polymer [KF6016 and the tradename (viscosity 150cSt, specific gravity 1.00, refractive index 1.448) by Shin-Etsu Chemical Co., Ltd.] was carried out, 5 weight sections addition of the following aramid fibers 1-3 was carried out, 25% silicone paste of a 2 and 5-dimethyl-screw (2, 5-tert-butyl peroxide) hexane was added with 2 weight sections and 2 rolls as a curing agent, and the wiper blade was cast using metal mold.

[0030] Aramid fiber 1: Para type aramid fiber (the Du Pont Kevlar, 2.0 deniers, 5mm)

Aramid fiber 2: Meta-mold aramid fiber (Conex by Teijin, Ltd., 2.0 deniers, 6mm)

Aramid fiber 3: Para-meta-mold aramid fiber (theque NORA by Teijin, Ltd., 2.0 deniers, 5mm)

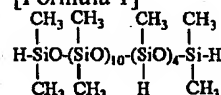
[0031] To the [example 4] above-mentioned base compound 1, the diatomaceous earth 20 weight section whose mean particle diameter is 3 micrometers is added with 2 rolls, and it is a hydrophilic grant agent further. 1.5 weight section addition of the polyether conversion dimethylsiloxane polymer (viscosity 150cSt, specific gravity 1.00, refractive index 1.448) was carried out, 5 weight sections addition of the aramid fiber 1 was carried out, 25% silicone paste of a 2 and 5-dimethyl-screw (2, 5-tert-butyl peroxide) hexane was added with 2 weight sections and 2 rolls as a curing agent, and the wiper blade was cast using metal mold.

[0032] To the [example 5] above-mentioned base compound 1, the diatomaceous earth 15 weight section whose mean particle diameter is 3 micrometers, and the diatomaceous earth 20 weight section whose mean particle diameter is 8 micrometers are added with 2 rolls, and it is a hydrophilic grant agent further. 1.5 weight section addition of the carbinol conversion dimethylsiloxane polymer [KF6003 and the tradename (viscosity 100cSt, specific gravity 0.98, a refractive index 1.408, functional-group equivalent 27 mgKOH/g) by Shin-Etsu Chemical Co., Ltd.] was carried out, 5 weight sections addition of the aramid fiber 1 was carried out, 25% silicone paste of a 2 and 5-dimethyl-screw (2, 5-tert-butyl peroxide) hexane was added with 2 weight sections and 2 rolls as a curing agent, and the wiper blade was cast using metal mold.

[0033] To the [example 6] above-mentioned base compound 1, the diatomaceous earth 15 weight section whose mean particle diameter is 3 micrometers, and the diatomaceous earth 15 weight section whose mean particle diameter is 8 micrometers are added with 2 rolls, and it is a hydrophilic grant agent further. 1.5 weight section addition of the polyether conversion dimethylsiloxane polymer (viscosity 150cSt, specific gravity 1.00, refractive index 1.448) was carried out, 5 weight sections addition of the aramid fiber 1 was carried out, the hydrogen siloxane shown by the chloroplatinic-acid content silicone-oil 0.1 weight section whose platinum concentration is 20 ppm as a curing agent, and the following formula was added with the 1.0 weight section and 2 rolls, and the wiper blade was cast using metal mold.

[0034]

[Formula 1]



[0035] As the fumed silica (product made from Japanese Aerosil) 35 weight section the organopolysiloxane 100 weight section whose average degree of polymerization it consists of 0.025 mol % of the 99.825 mol siloxane [0.15 mol % / of % and methyl vinyl siloxane units / and dimethyl vinyl] units of the [example 1 of comparison] dimethylsiloxane units, and is about 8,000, and whose specific surface area are 300m²/g, and a dispersant. It has a both-ends silanol group, the dimethylpolysiloxane 10 weight section whose viscosity in the average degree of polymerization of 13 or 25 degrees C is 15cs(es) was kneaded by the kneader, it heat-treated at 180 degrees C for 3 hours, and the base compound 2 was prepared.

[0036] To the above-mentioned base compound 2, the mean particle diameter added the diatomaceous earth 25 weight section which is 6 micrometers with 2 rolls, added 25% silicone paste of a 2 and 5-dimethyl-screw (2, 5-tert-butyl peroxide) hexane with 2 weight sections and 2 rolls as a curing agent, and cast the wiper blade using metal mold.

[0037] To the [example 2 of comparison] above-mentioned base compound 2, the diatomaceous earth 30 weight section whose mean particle diameter is 3 micrometers is added with 2 rolls, and it is a hydrophilic grant agent further. 1.5 weight section addition of the polyether conversion dimethylsiloxane polymer (viscosity 150cSt, specific gravity 1.00, refractive index 1.448) was carried out, 25% silicone paste of a 2 and 5-dimethyl-screw (2, 5-tert-butyl peroxide) hexane was added with 2 weight sections and 2 rolls as a curing agent, and the wiper blade was cast using metal mold.

[0038] To the [example 3 of comparison] above-mentioned base compound 2, the diatomaceous earth 30 weight section whose mean particle diameter is 8 micrometers is added with 2 rolls, and it is a hydrophilic grant agent further. 1.5 weight section addition of the polyether conversion dimethylsiloxane polymer (viscosity 150cSt, specific gravity 1.00, refractive index 1.448) was carried out, 25% silicone paste of a 2 and 5-dimethyl-screw (2, 5-tert-butyl peroxide) hexane was added with 2 weight sections and 2 rolls as a curing agent, and the wiper blade was cast using metal mold.

[0039] The physical properties of the silicone rubber which hardened the above-mentioned silicone rubber constituent and was obtained, and the mounting test result of each wiper blade are shown in Tables 1 and 2.

[0040]

[Table 1]

	実施例 1	実施例 2	実施例 3	実施例 4	実施例 5	実施例 6
Hs (JIS-A)	70	75	70	70	70	70
Ts (kgf/cm ²)	60	65	60	55	60	65
TR-A (kgf/cm)	40	35	35	40	34	45
実 装 試 験	ふき取り 性	良好	良好	良好	良好	良好
	びびり	なし	なし	なし	若干	若干
	滑り性	良好	良好	良好	良好	良好
	磨耗性	良好	良好	良好	良好	良好

[0041]

[Table 2]

	比較例 1	比較例 2	比較例 3
Hs (JIS-A)	70	75	70
Ts (kgf/cm ²)	65	80	50
TR-A (kgf/cm)	20	22	12
実 装 試 験	ふき取り 性	良好	良好
	びびり	若干	大
	滑り性	良好	悪い
	磨耗性	磨耗大	磨耗中

[0042] Measuring method Hs:JIS A type hardness meter by 6301 is used.

Ts:JIS 6301 TR-A:JIS 6301 wiping nature: It examined by having mounted in the real vehicle, and judged by viewing.

Chatter: It examined by having mounted in the real vehicle, and judged with viewing and sound.

Slipping nature: It examined by having mounted in the real vehicle, and judged by viewing.

Wear nature: After mounting for three months, it judged by viewing.

[0043]

[Effect of the Invention] According to the silicone rubber constituent of this invention, generally, a windshield wiper blade and the silicone wiper blade which has good slipping nature to any glass sides, and has sufficient tear strength especially are given.

[Translation done.]